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(54) Title of the Invention

ACRYLIC COPOLYMERS AND LEVELING AGENTS FOR POWDER COATING CONTAINING THE COPOLYMERS AS A COMPONENT

10 (57) Abstract: [Subject]

To provide a novel substance which is useful as a leveling agent for powder coating, and leveling agents for powder coating which contain said novel substance.

[Means of solution]

15 An acrylic copolymer, which is composed of (A) at least one kind of structural units of alkyl (meth)acrylate in which the alkyl group has 2-22 carbon atoms and alkenyl (meth)acrylate in which the alkenyl group has 2-22 carbon atoms, and (B) structural units of monocarboxylic acid- or monoamine-bound, isocyanate-modified acrylics and which has a number-average molecular weight of 4,000-100,000, is suitable as a component of leveling agents for powder coating.

[Claims]

[Claim 1] An acrylic copolymer which is characterized by being composed of (A) at least one kind of structural units of alkyl (meth)acrylate in which the alkyl group has 2-22 carbon atoms and alkenyl (meth)acrylate in which the alkenyl group has 2-22 carbon atoms, and (B) structural units of monocarboxylic acid-
5 or monoamine-bound, isocyanate-modified acrylics, and having a number-average molecular weight of 4,000-100,000.

[Claim 2] An acrylic copolymer which is characterized by being composed of the structural units (A) and (B) as described in Claim 1 and (C) structural units of (meth)acrylate having functional groups reactable with diisocyanate, and
10 having a number-average molecular weight of 4,000-100,000.

[Claim 3] An acrylic copolymer of Claim 2, which is characterized in that said structural unit (C) is of at least one kind selected from hydroxyalkyl (meth)acrylate, (the hydroxyalkyl group having 2-4 carbon atoms), alkyl monoalkylene glycol (meth)acrylate (the alkyl group having 1-12 carbon atoms),
15 alkenyl monoalkylene glycol (meth)acrylate (the alkenyl group having 1-12 carbon atoms), alkylalkenyl monoalkylene glycol (meth)acrylate (the alkylalkenyl group having 1-12 carbon atoms), alkyl polyalkylene glycol (meth)acrylate (the alkyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms), alkenyl polyalkylene glycol
20 (meth)acrylate (the alkenyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms) and alkylalkenyl polyalkylene glycol (meth)acrylate (the alkylalkenyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms).

[Claim 4] A leveling agent for powder coating, which is characterized by
25 comprising as a component a copolymer containing in its polymer chains (A) at least one kind of structural units of alkyl (meth)acrylate and alkenyl

(meth)acrylate, and (B) structural units of monocarboxylic acid- or monoamine-bound, isocyanate-modified acrylics.

5 [Claim 5] A leveling agent for powder coating as described in Claim 4, which is characterized in that said structural unit (B) is formed by modifying a functional group of a (meth)acrylate having a diisocyanate-reactable functional group with diisocyanate and binding thereto monocarboxylic acid or monoamine.

[Claim 6] A leveling agent for powder coating as described in Claim 4, which is characterized by comprising as a component the copolymer whose polymer chain comprises (C) structural units of (meth)acrylate having functional groups
10 reactable with diisocyanate.

[Claim 7] A leveling agent for powder coating as described in Claim 6, which is characterized in that said structural unit (B) is formed by modifying a functional group of a (meth)acrylate having a diisocyanate-reactable functional group with diisocyanate and binding thereto monocarboxylic acid or monoamine, and the
15 structural unit (C) is the (meth)acrylate whose functional group is left unmodified.

[Claim 8] A leveling agent for powder coating as described in Claim 4 or 6, which is characterized in that the copolymer has a solidifying point of at least 30°C.

20 [Claim 7 (sic)] A leveling agent for powder coating as described in Claim 4 or 6, which is characterized in that the weight ratio of the structural unit (A) to the structural unit (B) is 30-80 parts by weight to 70-20 parts by weight.

[Detailed Explanation of the Invention]

[0001]

25 [Technical field to which the invention belongs] This invention relates to novel acrylic copolymers and leveling agents comprising said copolymers as a

component.

[0002]

[Conventional art]

Non-aqueous paints, in particular, powder coating, in general are
5 inferior in leveling property and hence are difficult to be applied to usages
requiring high quality appearance. Recently, however, as a countermeasure
to environmental pollution, use of powder coating has been increasing to
replace solvent-based paints and demands for higher performance leveling
agents are also increasing incidentally thereto.

10 [0003] Thus, acrylic leveling agents have heretofore been proposed. For
example, JP9570/69 B described leveling agents suitable for
non-solvent-based liquid paint. As leveling agents for powder coating, for
example, JP84954/84A proposed mixture of amide wax with (meth)acrylate
copolymer.

15 [0004]

[Problem to be solved by the invention]

However, conventional leveling agents for powder coating give
markedly inferior painted appearance only, compared to the effect achieved
with use of leveling agents with solvent-based paints, although they exhibit a
20 recognizable extent of leveling effect. This is one reason for the limited utility
of powder coating.

[0005] This invention solves above problem in the conventional art and
provides a novel substance which is useful as leveling agent for powder coating.
The invention furthermore provides leveling agents for powder coating, which
25 comprise the novel substance as a component.

[0006]

[Means to solve the problem] We had anticipated usefulness of acrylic polymers as leveling agents for paint, in particular, powder coating, and under said anticipation synthesized various acrylic polymers and copolymers and conducted experiments to evaluate their performance as leveling agent for powder coating. Consequently, we have discovered novel acrylic copolymers exhibiting excellent performance and completed the present invention.

[0007] The acrylic copolymer as defined in Claim 1 of the present invention is characterized by being composed of (A) at least one kind of structural units of alkyl (meth)acrylate in which the alkyl group has 2-22 carbon atoms and alkenyl (meth)acrylate in which the alkenyl group has 2-22 carbon atoms, and (B) structural units of monocarboxylic acid- or monoamine-bound, isocyanate-modified acrylics, and having a number-average molecular weight of 4,000-100,000.

[0008] As the alkyl (meth)acrylate or alkenyl (meth)acrylate of the structural units (A), for example, methyl (meth)acrylate, ethyl (meth)acrylate, n-propyl (meth)acrylate, isopropyl (meth)acrylate, n-butyl (meth)acrylate, isobutyl (meth)acrylate, hexyl (meth)acrylate, 2-ethylhexyl (meth)acrylate, isodecyl (meth)acrylate, lauryl (meth)acrylate, cyclohexyl (meth)acrylate, stearyl (meth)acrylate, benzyl (meth)acrylate and isobornyl (meth)acrylate can be named. Of those, lauryl (meth)acrylate, butyl (meth)acrylate and 2-ethylhexyl (meth)acrylate are preferred for the use as leveling agents for powder coating.

[0009] As the isocyanate-modified acrylics of structural unit (B), for example, 2-isocyanate ethyl methacrylate and methacroyl isocyanate can be named. Furthermore, the monocarboxylic acid to bind to the isocyanate-modified acrylics of structural unit (B) preferably contain at least 12 carbon atoms, examples of which include lauric acid, myristic acid, palmitic acid, stearic acid,

behenic acid and benzoic acid. As the monoamine, primary or secondary monoamines are preferred, examples of which include laurylamine, stearylamine, oleylamine, benzylamine, aniline and distearylamine.

[0010] The acrylic copolymer as defined in Claim 2 is characterized by being composed of said structural units (A) and (B) and (C) structural units of (meth)acrylate having functional groups reactable with diisocyanate, and having a number-average molecular weight of 4,000-100,000.

[0011] Said structural unit (C) is, for example, of at least one kind selected from hydroxyalkyl (meth)acrylate, (the hydroxyalkyl group having 2-4 carbon atoms), alkyl monoalkylene glycol (meth)acrylate (the alkyl group having 1-12 carbon atoms), alkenyl monoalkylene glycol (meth)acrylate (the alkenyl group having 1-12 carbon atoms), alkylalkenyl monoalkylene glycol (meth)acrylate (the alkylalkenyl group having 1-12 carbon atoms), alkyl polyalkylene glycol (meth)acrylate (the alkyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms), alkenyl polyalkylene glycol (meth)acrylate (the alkenyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms) and alkylalkenyl polyalkylene glycol (meth)acrylate (the alkylalkenyl group having 1-12 carbon atoms and the polyalkylene glycol group having 2-4 carbon atoms).

[0012] Specific examples of said hydroxyalkyl (meth)acrylate include 2-hydroxyethyl (meth)acrylate, 2-hydroxypropyl (meth)acrylate and 2-hydroxybutyl (meth)acrylate. Specific examples of said alkyl (or alkenyl or alkylalkenyl) mono (or poly) alkylene glycol (meth)acrylate include methoxydiethylene glycol (meth)acrylate, methoxypropylene glycol (meth)acrylate, n-butoxyethylene glycol (meth)acrylate, 2-phenoxyethyl (meth)acrylate, trioxyethylenenonylphenol (meth)acrylate, acetoacetoxethyl

(meth)acrylate, 2-hydroxy-3-phenoxypropyl (meth)acrylate and 2-(meth)acryloyloxyethyl-2-hydroxyethyl-phthalic acid.

[0013] The leveling agent for powder coating of the present invention as described in Claim 4 comprises as a component a copolymer containing in its
5 polymer chains (A) at least one kind of structural units of alkyl (meth)acrylate and alkenyl (meth)acrylate, and (B) structural units of monocarboxylic acid- or monoamine-bound, isocyanate-modified acrylic.

[0014] As described in Claim 5, the leveling agent for powder coating as defined in Claim 4 can be favorably worked, where the structural unit (B) is a
10 (meth)acrylate formed by modifying a functional group of a (meth)acrylate having a diisocyanate-reactable functional group with diisocyanate and binding thereto monocarboxylic acid or monoamine. The functional group reactable with diisocyanate is, for example, hydroxyl, and it is sufficient that at least one of such functional group is present in the (meth)acrylate molecule. After reacting
15 this functional group with diisocyanate, the product is reacted with monocarboxylic acid or monoamine by a means known per se.

[0015] The leveling agent for powder coating as described in Claim 6 of the present invention comprises, in the polymer chains of the copolymer, said structural units (A) and (B), and further the structural unit (C) of (meth)acrylate
20 having functional group(s) reactable with diisocyanate.

[0016] As described in Claim 7, where the structural unit (B) is a (meth)acrylate formed by modifying a functional group of a (meth)acrylate having a diisocyanate-reactable functional group with diisocyanate and binding thereto monocarboxylic acid or monoamine, and the structural unit (C) is the
25 (meth)acrylate whose functional group(s) are left unmodified, the leveling agent for powder coating as described in Claim 6 can be favorably worked.

[0017] As examples of the diisocyanate which is adequate for reacting with the (meth)acrylate having functional group(s) reactable with diisocyanate include: tolylene diisocyanate, 4,4'-diphenylmethane diisocyanate, xylylene diisocyanate, methaxylylene diisocyanate, 4,4'-methylenebis-

5 (cyclohexylisocyanate), isophorone diisocyanate, trimethylhexamethylene diisocyanate, 1,5-naphthalene-2-diisocyanate, tolylene diisocyanate (sic) and dimerized polymers of these isocyanates. Of these, 2,4-tolylene diisocyanate is preferred because of the ease in controlling the reaction.

[0018] For leveling agents for powder coating of the present invention, the
10 copolymers preferably have a solidifying point not lower than 30°C, as specified in Claim 8. Still more advantageously, it is not lower than 40°C. Where the solidifying point is not lower than 30°C, [the copolymer] is readily powderized and hence is easily mixed into powder coating. Whereas, when it is lower than 30°C, blending of a carrier such as silica, a wax or the like becomes necessary
15 at the powderization time. Such blending material(s) may further call for care to prevent side effect or further increase in [the copolymer's] use rate.

[0019] The leveling agent for powder coating of the present invention can be advantageously worked when the weight ratio of the structural unit (A) to the structural unit (B) is within a range of 30-80 parts by weight to 70-20 parts by
20 weight, as specified in Claim 9.

[0020] The copolymers according to the present invention are obtainable by copolymerizing one or more kinds of monomers of the structural unit (A) with a monomer of the structural unit (B). As a different means, the object copolymer can also be acquired by copolymerizing monomer(s) of the structural unit (A)
25 with an isocyanate-modified acrylic monomer which is an intermediate starting material of the structural unit (B), and then reacting with the diisocyanate

groups in the formed copolymer with monocarboxylic acid or monoamine.

Furthermore, the object copolymer can also be obtained by copolymerizing monomer(s) of the structural unit (A) with (meth)acrylate monomer having a functional group reactable with diisocyanate in its molecule, which is an

5 intermediate starting material of the structural unit (B) monomer; isocyanatizing the formed copolymer to convert it to a copolymer having isocyanate groups; and reacting the diisocyanate groups with monocarboxylic acid or monoamine.

[0021] Leveling effect of the leveling agents for powder coating according to the present invention is affected by molecular weight of the copolymer. [The
10 copolymer is, therefore,] synthesized to have a molecular weight within a range of 4,000-100,000, preferably 6,000-50,000. Use of a polymerization regulating agent such as alkyl mercaptan or the like in that occasion allows easier molecular weight control.

[0022] Said copolymerization can be conducted by any known polymerization
15 method. For example, solution polymerization method is generally practiced, by continuing the reaction for about 1-15 hours, preferably 4-8 hours, normally at 70-150°C, preferably 80-130°C, in the presence of a polymerization initiator.

[0023] It is desirable to use, as the reaction solvent, one which is capable of dissolving the copolymer formed, and which can be easily removed after the
20 polymerization by such means as condensation under reduced pressure. As such a solvent, aromatic solvents such as toluene, xylene, Solvesso 100 and the like, and acetic acid esters such as butyl acetate, isobutyl acetate and the like are desirable. As the polymerization initiator, for example, azo-compounds such as azobisisobutyronitrile, dimethyl azobisisobutyrate and
25 the like, and organic peroxides such as lauroyl peroxide, diisopropyl benzene hydroperoxide and the like, which are radical initiators useful for ordinary radical

polymerization, can be used.

[0024] Furthermore, the leveling agents for powder coating according to the present invention can give favorable appearance to coated film formed by paint application, by concurrent use of de-gasing agent, e.g., benzoin, with the copolymer. It is also permissible to adsorb the polymer onto silica or mix it with wax such as hydrogenated castor oil in the occasion of adding the copolymer to powder coating as a leveling agent, for facilitating the addition. Still other additive(s) may be added.

[0025]

10 [Effect of the invention]

According to the invention, novel acrylic copolymers having a use as leveling agents for powder coating are obtained. Mixing of such an acrylic copolymer into powder coating is effective for adjusting flowability of the coating during the period from immediately after the coating application until hardening of the coating, and for improving smoothness and gloss of the coated surface after completion of the painting.

15 [0026] Leveling agents for powder coating according to the invention which contain said acrylic copolymer as a component give, when added to powder coating and applied onto coating objects, coated surfaces exhibiting excellent leveling property and gloss. The leveling agents furthermore have no detrimental effect on performance of the coating film on the coated object. Still in addition, the leveling agents for powder coating according to the present invention can be readily powderized and hence can be easily added to powder coating.

25 [0027]

[Examples] The present invention is explained in further details, referring to

the following Examples. The invention, however, is not limited to these Examples.

[0028] Example 1

Three-hundred (300) parts of butyl acetate was fed into a polymerizer equipped with a reflux condenser, thermometer, stirrer and dropping tank, and into which a solution of a mixture of the following composition was dropped in nitrogen atmosphere at 110°C, consuming about 3 hours. After completion of the dropping, 5 parts of azobisisobutyronitrile was added and the system was maintained at 110°C for 2 hours.

10 [0029]

stearyl acrylate	324 parts
methacryloyl isocyanate	111 parts
dodecyl mercaptan	10 parts
azobisisobutyronitrile	15 parts
butyl acetate	200 parts

15

Then 281 parts of stearic acid was added and reacted at 110°C for 3.5 hours. Then the temperature was raised to 130°C and butyl acetate was distilled off under reduced pressure, to provide the object copolymer.

[0030] This copolymer had a number-average molecular weight of 9,300,

20

when analyzed of its molecular weight distribution with a Gel Permeation Chromatography (GPC) Analyzer (Showa Denko K.K., Snodex GPC System).

According to infrared absorption spectral analysis, the absorption band at 2200 cm^{-1} disappeared to confirm disappearance of the isocyanate group. Those

absorption bands corresponding to amide group $-\text{CONH}-$ at 3310 cm^{-1} , 1645

25

cm^{-1} and 1550 cm^{-1} were present; and also the absorption band at 1735 cm^{-1}

which is attributable to the ester group in the acrylate also was present. Thus

synthesis of the intended copolymer was confirmed. By the analysis with Differential Scanning Calorimeter DSC 8230B, manufactured by Rigaku Denki Kabushiki Kaisha, an endothermic peak was observed at 48.9 °C, allowing confirmation of the solidifying point.

5 [0031] Example 2

Under the conditions identical with those in Example 1, 300 parts of butyl acetate was fed and copolymerization of a blend of monomers and initiator of the following composition was conducted, and the formed copolymer was reacted with 170 parts of behenic acid to provide a copolymer.

10 [0032]

2-ethylhexyl acrylate	184 parts
2-methacryloyloxyethylisocyanate	78 parts
dodecyl mercaptan	10 parts
azobisisobutyronitrile	2 parts

15 According to the analyses similar to those conducted in Example 1, the copolymer had a number-average molecular weight of 34,000. Disappearance of isocyanate groups and presence of amide and ester groups were observed, confirming synthesis of the object copolymer. The solidifying point was 43.5°C.

20 [0033] Example 3

Using an apparatus similar to that used in Example 1, 300 parts of butyl acetate was fed, the blend of monomers and initiating agent was given the following composition and the monomers were polymerized under the same conditions with Example 1.

25 [0034]

stearyl acrylate	324 parts
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2-hydroxyethyl acrylate	58 parts
dodecyl mercaptan	10 parts
azobisisobutyronitrile	2 parts

5 Cooling the system to 40°C, 32 parts of tolylene diisocyanate was added, followed by 2 hours' reaction and further by 2 hours' reaction at an elevated temperature of 80°C.

[0035] Then 57 g of stearic acid was added, followed by 2 hours' reaction at 100°C. The temperature was further raised to 130°C and butyl acetate was distilled off under reduced pressure, to provide the intended copolymer.

10 [0036] According to the analyses similar to those in Example 1, the copolymer had a number-average molecular weight of 4,000. Absorption bands attributable to urea linkage -NHCONH- were present at 3320 cm^{-1} , 1630 cm^{-1} and 1560 cm^{-1} , allowing confirmation of synthesis of the object copolymer. The solidifying point was 34.8°C.

15 [0037] Example 4

Using an apparatus similar to that used in Example 1, 300 parts of butyl acetate was fed, the blend of monomers and initiating agent was given the following composition and the monomers were polymerized under the same conditions with Example 1.

20 [0038]

stearyl acrylate	324 parts
2-hydroxyethyl acrylate	58 parts
dodecyl mercaptan	10 parts
azobisisobutyronitrile	2 parts

25 Cooling the system to 40°C, 32 parts of tolylene diisocyanate was added, followed by 2 hours' reaction and further by 2 hours' reaction at an

elevated temperature of 80°C (operations up to this were same as Example 3).

[0039] To the system then 100 parts of distearylamine was added and reacted at 100°C for 2 hours. The temperature was raised to 130°C and the butyl acetate was distilled off under reduced pressure to provide a copolymer. This copolymer had a solidifying point of 43.8°C.

[0040] Comparative Example 1

Under the same conditions as those in Example 1, a monomeric blend of the following composition was processed to give a copolymer having a number-average molecular weight of 18,000:

10 [0041]

2-ethylhexyl acrylate	50 parts
dodecyl mercaptan	2.5 parts
azobisisobutyronitrile	1 part.

Comparative Example 2

15 Under the same conditions as those in Example 1, a monomeric blend of the following composition was processed to give a copolymer having a number-average molecular weight of 31,000:

[0042]

2-ethylhexyl acrylate	45 parts
20 methoxydiethylene glycol methacrylate	5 parts
dodecyl mercaptan	0.5 part
azobisisobutyronitrile	1 part.

Comparative Example 3

25 BYK 360P (an acrylic polymer adsorbed onto a special silica, a leveling agent for powder coating manufactured by Bic Chemie (phonetic) Co.) was used.

[0043] Comparative Example 4

Hydrogenated castor oil (castor wax) was used.

[0044] Products of above Examples and Comparative Examples were used as leveling agents and subjected to performance tests. As the powder coating
5 into which each leveling agent was mixed, a polyester urethane powder coating was used.

[0045] The composition of coating blend was as follows:

[0046]

	① Finedick (phonetic) M-8020	82 parts
10	② Crelan (phonetic) U1	15 parts
	③ Epichrone (phonetic) 2055	3 parts
	④ titanium oxide	50 parts
	⑤ benzoin	0.3 part
	⑥ leveling agent	2.5 parts
15	<hr/>	
	Total	152.8 parts

① Finedick M-8020: a polyester resin, manufactured by Dainippon Ink
20 Kagaku Kogyo

② Crelan U1: a blocked isocyanate, manufactured by Sumitomo Bayer
Urethane Co.

③ Epichrone 2055: an epoxy resin, manufactured by Dainippon Ink
Kagaku Kogyo

25 ④ Tipake (phonetic) CR-93: product of Ishihara Sangyo

⑥ leveling agent: Examples 1-4, and Comparative Examples 1-4

Method of addition and paint formulation

[Each of] above blends was thoroughly mixed in a Henschel mixer

(Mitsui Miike Kako) and further melt-kneaded with Twin-screw Extruder Model MP-2015 (APV Chemical Machinery Co.), at a barrel temperature of 100°C. The kneaded product was cooled, recovered in fractions, further ground with a grinder and sieved through a wire net of 105 μm in mesh size, and the powder which passed through the meshes was used as the coating for performance evaluation.

[0047] Method for preparing (coating) test samples

About 8 g of the coating was applied onto a degreased SPCC-B dull steel sheet (100 mm \times 200 mm) with an electrostatic coater (GX-3300S, Onoda Cement Co.), baked and hardened at 190°C for 20 minutes to provide a sample for performance tests.

[0048] Test methods

In the gloss test, gloss of the coating film on each of the samples for performance test as prepared by the above method was measured at 60°C and 20°C with a digital glossmeter (Murakami Shikisai Co.). Leveling property (smoothness) was evaluated by visual observation of the coating film of each sample and concurrently, center line average roughness R_a , maximum height R_{max} and crossing (+) point average roughness R_z (all in accordance with JIS B0601-1982) were determined with a surface roughness form measuring device Surfcom 590-3D (Tokyo Seimitsu Co.). Also surface damage was evaluated by visual observation of each sample and counting the number of dewetting.

[0049] Test result

The results of the tests given to the samples by the above test methods are shown in the following Table 1. As can be understood from Table 1, in respect of all the test items of gloss, surface damage (dewetting number) and

leveling property, the performance test samples in which the leveling agents obtained in the working Examples 1-4 of the present invention excelled over those in which the leveling agents from Comparative Examples 1-4 not following the present invention were used and the blank (powder coating without using a leveling agent).

[0050]

[Table 1]

Table 1. Test Results

Leveling Agent	Surface Damage (number of dewetting)	60° Gloss	20° Gloss	Leveling Property		
				R _a	R _{max}	R _z
Blank	Many	56.4	16.1	0.182	14.2	9.5
Example 1	None	99.0	93.0	0.031	2.1	1.3
Example 2	None	93.0	78.0	0.051	2.5	1.8
Example 3	None	92.6	76.0	0.041	2.8	2.0
Example 4	None	93.0	78.0	0.042	2.4	2.1
Comp. Example 1	None	85.3	51.0	0.157	6.2	3.3
Comp. Example 2	None	85.0	50.1	0.160	7.4	3.8
Comp. Example 3	1-2	91.1	50.5	0.088	2.9	2.5
Comp. Example 4	5-10	77.7	47.6	0.150	10.5	5.8